Doping method – properties relations in niobium doped titanium dioxide

Stanislav Simakov, Sioma Baltianski and Yoed Tsur

Chemical Engineering Department

Technion – Israel Institute of Technology

Niobium doped titanium dioxide has been prepared with various Nb concentrations by two different routes. The first was co-precipitation of titanium and niobium salts in order to create titanium dioxide nanoparticles with a uniform distribution of the dopant within them. The second was incorporation of Nb into already prepared titania, to create a spatial distribution of Nb within the nanoparticle.

A number of methods were used for detailed characterization of the prepared nanopowders. X-ray diffraction was used for crystalline structure determination. Scanning electron microscopy observations were performed for the powder morphology investigation. Differential scanning calorimetry was performed to investigate reversible and irreversible enthalpic transitions during thermal treatments. Dynamic light scattering was used for particle size determination. Electrophoretic light scattering and phase analysis light scattering were performed to investigate the zeta potential in liquid medium. In order to determine the distribution of the dopant (niobium) concentration within a single nanoparticle, Auger electron spectroscopy combined with in-situ ion beam sputtering was performed. Electrical properties were measured using impedance spectroscopy in a wide range of frequency and at different temperatures. The possibility of creation of a core-shell structure in such systems is discussed, considering the experimental results.

Highlights from the presentation

Both routes of preparation resulted in quite homogenous concentration of Nb within the grains. The co-precipitation route resulted in higher solubility of Nb. Even at concentrations of Nb/Ti=0.25, we find solid solution with no second solid phase and very little segregation of Nb to the grain boundaries. See Figure 1 below:

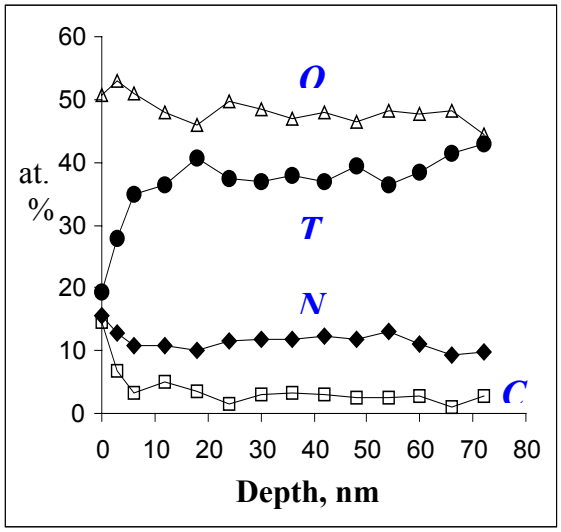

Figure 1: elements distribution by AES within the anatase nanoparticles prepared by coprecipitation with Nb/Ti=0.25, calcined at 760ºC. (D~100nm).

The shell incorporation route resulted, at very high concentrations of Nb, in two solid phases. The Nb concentration in titania was homogenous and a second niobium oxide phase doped with some homogenously distributed Ti was formed. An example of the titania phase and the concentration distributions is shown in Figure 2 below. The second Nb-rich phase is shown in figures 3 and 4. This suggests that the incorporation of Nb to the surface of the titania is a rate-limiting step.

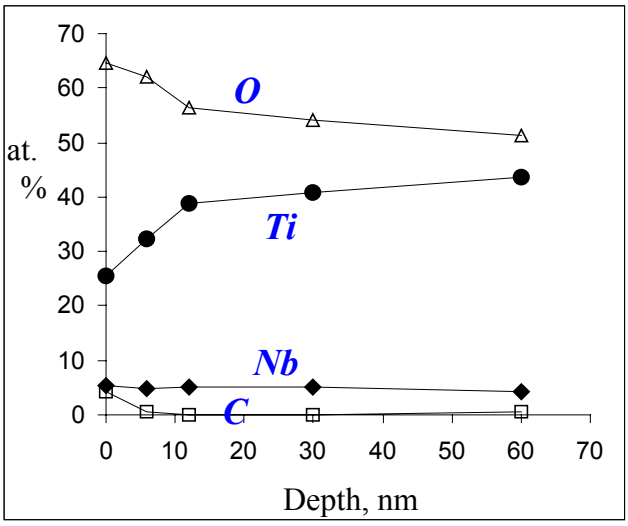

Figure 2: elements distribution by AES within the anatase nanoparticles prepared by shell incorporation with Nb/Ti=0.25, calcined at 760ºC. (D~100nm).

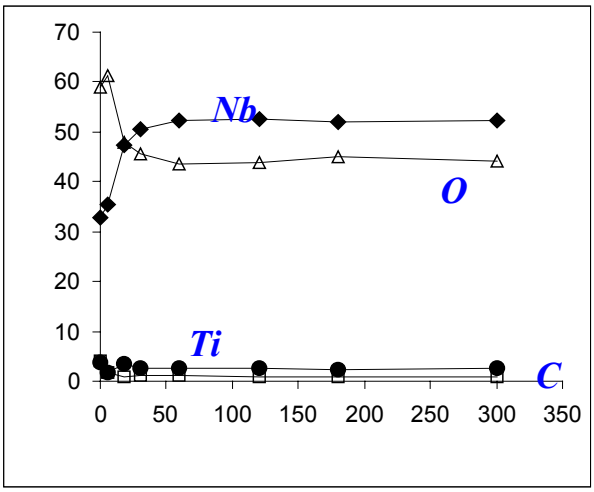

Figure 3: elements distribution by AES within the Nb-rich phase, calcined at 760ºC. (D~1μm).

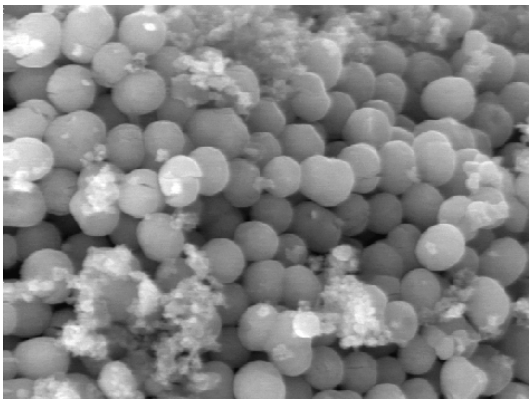

Figure 4: SEM image of the niobium oxide phase (D~1μm).